

Monthly Report

Contract Number: N00014-15-C-0087

Grant: ONR 25-1109-0140-001

Project Title: Portable Fiber Laser System and Method to Remove Pits and Cracks on Sensitized Surfaces of Aluminum Alloys

Prepared for
DEPARTMENT OF THE NAVY
Office of Naval Research

For the period
July 1, 2015, to July 30, 2015

Submitted by
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Report Documentation Page				Form Approved OMB No. 0704-0188	
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1. REPORT DATE AUG 2015		2. REPORT TYPE		3. DATES COVERED 01-07-2015 to 30-07-2015	
4. TITLE AND SUBTITLE Portable Fiber Laser System and Method to Remove Pits and Cracks on Sensitized Surfaces of Aluminum Alloys				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University of Nebraska-Lincoln,,Department of Electrical and Computer Engineering,,209N Walter Scott Engineering Center,,Lincoln,,NE,08588				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT The goal of this project is to develop a compact, portable fiber laser repair system integrated with the capabilities of surface cleaning, local heating, and peening using a single laser to remove pits and cracks in sensitized Al alloy surfaces and to condition those surfaces to improve resistance to further sensitization. The fiber laser repair system will have the advantages of being compact, portable, energy saving, chemical free, maintenance free, and easy to operate meeting the U.S. Navy???s goal of reducing operation, maintenance, and support costs associated with 5000 series Al sensitization.					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT Same as Report (SAR)	18. NUMBER OF PAGES 17	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

Abstract

The **goal** of this project is to develop a compact, portable fiber laser repair system integrated with the capabilities of surface cleaning, local heating, and peening using a single laser to remove pits and cracks in sensitized Al alloy surfaces and to condition those surfaces to improve resistance to further sensitization. The fiber laser repair system will have the advantages of being compact, portable, energy saving, chemical free, maintenance free, and easy to operate meeting the U.S. Navy's goal of reducing operation, maintenance, and support costs associated with 5000 series Al sensitization.

During this reporting period (July 1, 2015 to July 30, 2015), the experimental setup for laser shock peening (LSP) was developed. The effects of LSP on the surface topography, nanohardness, and elastic modulus of the 5052 Mg-Al alloy samples were experimentally investigated and analyzed. The phased objectives and specific content are as follows:

- (1) Selection and Purchase of 5000 series Al alloys and temperature-controlled oven for sensitization experiment.
- (2) Developing the experimental setup for laser shock peening.
- (3) Optimization of processing parameters for laser shock peening.
- (4) Surface morphology study of the laser-peened surfaces using optical microscopy and scanning electron microscopy.
- (5) Study of the surface nanohardness and elastic modulus of the laser-peened 5052 Al alloy samples using the nanoindentation technique.

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1. Usage of Project Funding

Total contract amount: \$549,999 (current year).

Cost incurred during the performance period to date: \$2,674.

Costs incurred to date: \$2,674.

A financial statement is attached at the end of the project. During this reporting period, the purchase of some equipment and supplies is planned; they are listed below. A new postdoc is being hired and will join the project in September 2015.

Table 1. Equipment and supplies to be purchased.

No.	Instruments	Manufacturer	Main parameters	Price (\$)
1	Forced Air Oven	SHEL LAB	Temperature range 40-250°C Temperature uniformity $\pm 1.5^{\circ}\text{C}$ @150°C	2,719.92
2	Medium Forced Air Incubator	VWR	Temperature range 5-75°C Temperature stability $\pm 0.1^{\circ}\text{C}$ @37°C Temperature uniformity $\pm 0.3^{\circ}\text{C}$ @37°C	3039.11
3	Scientific Precision™ General-Purpose Water Baths	Fisher Scientific	Temperature range ambient-99°C Temperature stability $\pm 0.1^{\circ}\text{C}$ @37°C Temperature uniformity $\pm 0.2^{\circ}\text{C}$ @37°C	693.55
4	5083-H116 Al-Mg alloy materials	McMaster Carr	Strengthened corrosion-resistant 5083-H116 aluminum, sheet, 1/4" thick, 2" x 24", 2 pieces	71.60
5	Reagent	VWR & Fisher	Nitric acid and sodium hydroxide for mass loss test, other acids for etching, electrochemical polishing, and anodizing	700.28
6	Containers	VWR	Beakers, petri dishes, bottles, graduated cylinders	425.26

2. Schedule and Current Completion Rate of Milestones

The **goal** of this project is to develop a compact, portable fiber laser repair system integrated with the capabilities of surface cleaning, local heating, and peening using a single laser to remove pits and cracks in sensitized Al alloy surfaces and to condition those surfaces to improve resistance to further sensitization. The fiber laser repair system will have the advantages of being compact, portable, energy saving, chemical free, maintenance free, and easy to operate meeting the U.S. Navy's goal of reducing operation, maintenance, and support costs associated with 5000 series Al sensitization. The major goals of this project include four phases/objectives:

- **Phase 1:** Laser cleaning to remove surface contamination on pits and cracks in sensitized regions.
- **Phase 2:** Laser peening of sensitized 5000 series Al alloys.
- **Phase 3:** Surface evaluation after laser cleaning and peening.
- **Phase 4:** Development of a field-portable desensitization repair system.

Table 2 summarizes the current progress of each phase/objective during this reporting period.

Table 2. The completion rate for each phase/objective of the project.

Phases	Major goals and milestones	Starting date (mm/dd/yy)	Ending date (mm/dd/yy)	Completion rate (%)
Phase 1	1) Experimental setup for laser cleaning developed	5/15/2015	5/14/2016	0
	2) Cleaning mechanism and parameter windows	11/15/2015	4/29/2017	0
	3) Real-time monitoring of cleaning process	11/15/2015	4/29/2017	0
	4) Laser cleaning using fiber laser	11/15/2015	4/29/2017	0
Phase 2	1) Sensitization of Al alloys	5/15/2015	4/29/2017	0
	2) Experimental setup for laser peening developed	5/15/2015	5/14/2016	20
	3) The effect of different coated layers	11/15/2015	4/29/2017	20
Phase 3	1) DoS for Al samples after laser cleaning and peening obtained	11/15/2015	4/29/2018	0
	2) Pitting and crack and strength after laser cleaning and peening analyzed	11/15/2015	4/29/2018	0
Phase 4	1) Configuration and design	4/30/2017	10/31/2017	0
	2) Selection and test of fiber laser	11/1/2017	4/29/2019	0
	3) Selection, test, and program a galvano scanner	11/1/2017	4/29/2019	0
	4) Electrical, optical, mechanical integration	4/30/2018	4/29/2019	0
	5) System specifications and field-portable test	4/30/2018	4/29/2019	0

3. Progress and Major Accomplishments

3.1 Methods and Procedures

3.1.1 Basic Process

The basic process of laser shock peening (LSP) is illustrated in Fig. 1. LSP is a cold mechanical process where pressure waves caused by expanding plasma plastically deform the surface of a material. LSP uses a thin layer of ablative material that is opaque to the laser. The opaque ablative material, typically black spray paint or tape, was used as a sacrificial layer in the early

study by Fairland and Clauer [1]. The sacrificial layer also minimizes thermal effects on the surface caused by the laser. The laser partially vaporizes the ablative layer to form high-pressure plasma. The plasma, confined by a thin film of water (confining medium), expands rapidly resulting in a recoiling pressure wave on the order of GPa, as reported by Fariland et al. and Caslaru, et al. [2-3]. The pressure wave is a cold mechanical process that typically deforms the surface. The plasma-induced shock pressure causes severe plastic deformation, refined grain size, compressive residual stresses, and increased hardness at the surface and in the subsurface. As a result, the mechanical properties on the workpiece surface will be enhanced to improve corrosion performance and damage by a foreign object.

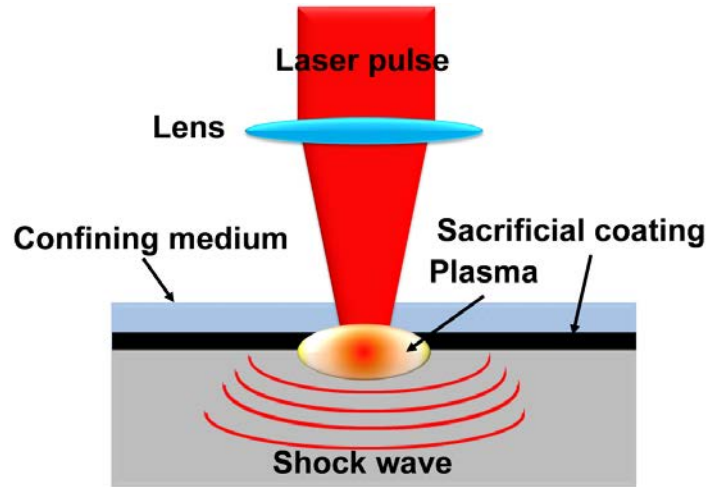


Figure 1. Schematic of the principle of LSP.

Table 3. Chemical compositions of the tested materials (wt.%).

Material	Mg	Si	Cr	Mn	Fe	Al
5052 Al alloy	2.68	0.42	0.41	0.24	---	Remainder

Table 4. Mechanical properties of the tested materials.

Material	Yield strength (kgf mm ⁻²)	Tensile strength (kgf mm ⁻²)	Elongation (%)	Specific gravity (g cm ⁻³)
5052 Al alloy	193	228	18	2.68

3.1.2 Material Preparation

During this reporting period, the samples tested were prepared from 5052 Al alloy plate, which was cut into rectangular shapes with approximate dimensions of 30 mm × 30 mm × 2 mm (length × width × thickness). The chemical composition and mechanical properties of 5052 Al alloy are shown in Tables 3 and 4, respectively. Prior to the LSP treatment, the samples were polished mechanically with sand paper of different grades of roughness to achieve a mirror-like

surface, enabling the nanoindentation test to be conducted, which requires low surface roughness of several hundred nanometers. The polishing was followed by ultrasonic cleaning, first with acetone and then deionized water, to degrease the sample surface. All samples were prepared shortly before the laser peening experiments.

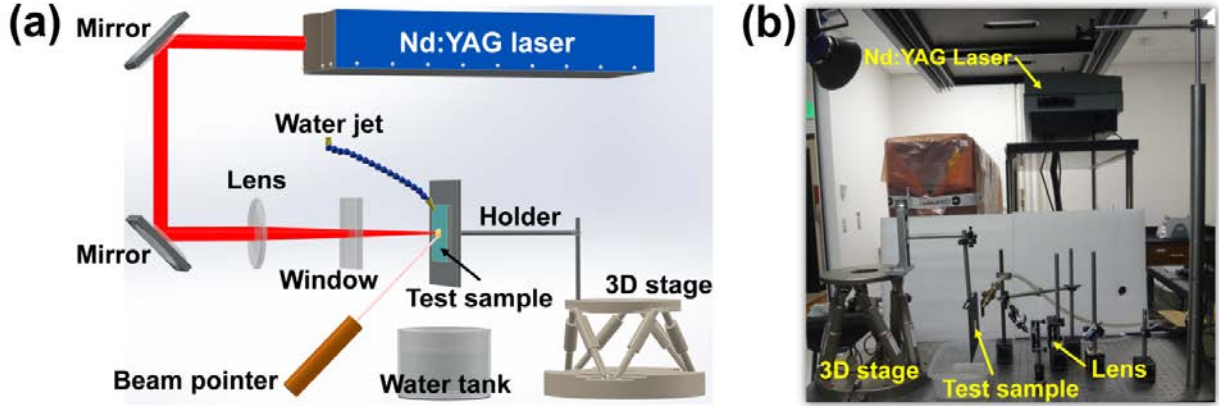


Figure 2. (a) Schematic diagram of the experimental setup for LSP. (b) LSP system established in our lab.

3.1.3 Experimental Setup

Figure 2a shows the schematic diagram of the experimental setup for LSP. The LSP system developed in our lab is shown in Fig. 2b. This laser peening system can meet the demands of single and multiple LSP impacts. The laser source is a continuum Q-switched Nd:YAG PR II 8010 laser. The laser pulse frequency can be changed from 1 to 10 Hz. The pulse duration normally used with this laser is approximately 6-8 ns, and the maximum pulse energy is 850 mJ with a wavelength of 1064 nm. The output diameter of the laser beam from this laser is about 5 mm.

Two flat mirrors and a flat convergent lens (focal length: 20 cm) were used to deliver the pulse produced by the laser. The samples were placed vertically at the focal plane. The size of the laser spot focused on the samples measured approximately 1 mm.

In this LSP system, purified water was used as the confining medium. Control of water purity is important in order to avoid the formation of water bubbles or the concentration of impurities coming from the material ablation due to the laser irradiation. The appearance of suspended elements can affect the laser peening process by their interaction with the pulsed laser beam. Continuous application of water is a good solution to avoid these elements. Therefore, a special device for producing controlled water jets was implemented to form a thin layer of water on the sample surface to be treated. By controlling the water velocity and jet direction, it was possible to produce a water layer with a constant thickness. Al foil tape and flat black tape were chosen for the sacrificial layers. Before the LSP experiment, the sample surfaces were covered with Al foil

or a layer of black tape with thicknesses of 100 and 177 μm , respectively. The sample prepared was fixed on a holder and mounted on a motor-controlled, five-axis work stage with five degrees of freedom, a resolution of 1 μm , and a maximum speed of 20 mm/s. The processing parameters of laser peening are summarized in Table 5. During this reporting period, a single-spot LSP experiment was carried out to test the effectiveness of the LSP system established. We used a laser source with a frequency of 1 Hz for conveniently controlling the laser impact times on one point.

Table 5. The processing parameters used in the laser peening experiment.

Laser system	Continuum Nd:YAG PR II 8010 laser; wavelength: 800 nm; pulse energy: 650 mJ; pulse duration: 6-8 ns; frequency: 1-10 Hz; beam size: ~ 1 mm
Confining medium	Flowing water with layer thickness of approximately 1-2 mm
Sacrificial coating	(1) No coating; (2) Al foil tape (100 μm); (2) flat black tape (177 μm)

3.1.4 Characterization

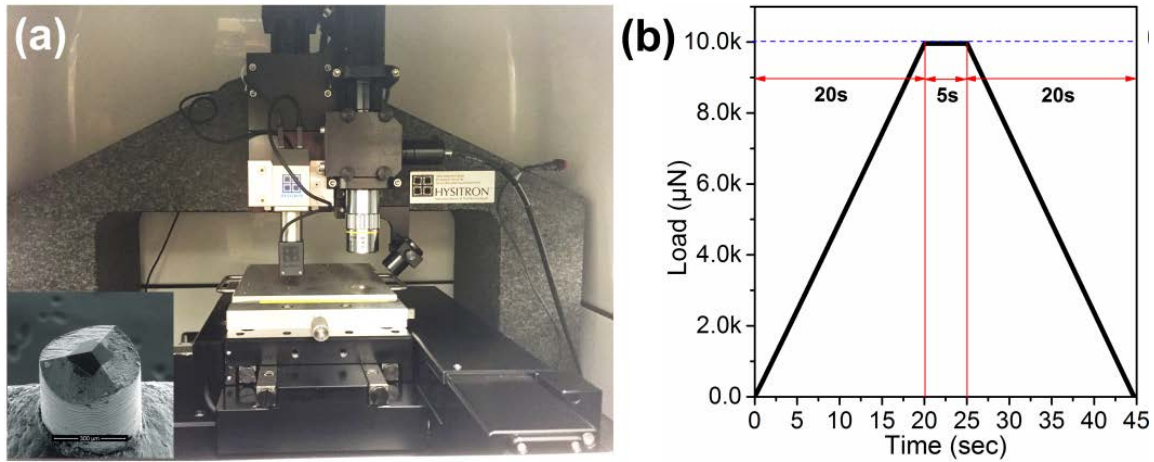


Figure 3. (a) An optical image of the nanoindenter used for mechanical property tests. The inset in (a) shows a typical SEM image of the nanomechanical testing probe used in our experiment. (b) Load-time $F = F(t)$ function for time-dependent indentation.

After laser peening, the surface quality of the processed samples was systematically investigated. Various analytical techniques were used for characterization of the samples before and after laser peening for optimizing the laser peening parameters. Optical microscopy (Nikon ECLIPSE, Japan) and scanning electron microscopy (SEM, Philips, XL 30E, 5kV) were used to study the morphology of the laser-peened samples. A 3D profiler (New View 8000, Zygo Corporation) was used to measure the 3D profiles of the macro plastic deformation from the bottom surface of

the samples. The micromechanical properties of all samples tested were carried out using nanoindentation (Hysitron nanoindenter) (Fig. 3a). All nanoindentation measurements were performed with the standard three-sided pyramidal Berkovich (TI-00039) probe. In all nanoindentation tests, a total of five indents were measured and averaged to determine the mean surface nanohardness (H) and elastic modulus (E) values for statistical purposes. In the 5052 Al alloy nanoindentation experiment, the loading rate, maximum applied load, and loading time were 500 $\mu\text{N/s}$, 10000 μN , and 20/5/20 s, respectively (Fig. 3b).

3.2 Results and Discussion

3.2.1 Optimization of the Processing Parameters of Laser Peening Using 5052 Al Alloy

The important processing parameters related to LSP conditions are laser processing parameters, the confining medium, and sacrificial coatings, which can significantly affect the mechanical properties of the alloys and metallic materials. The objective of this task was to investigate the effectiveness of the established laser peening system using 5052 Al alloy for the samples to be tested.

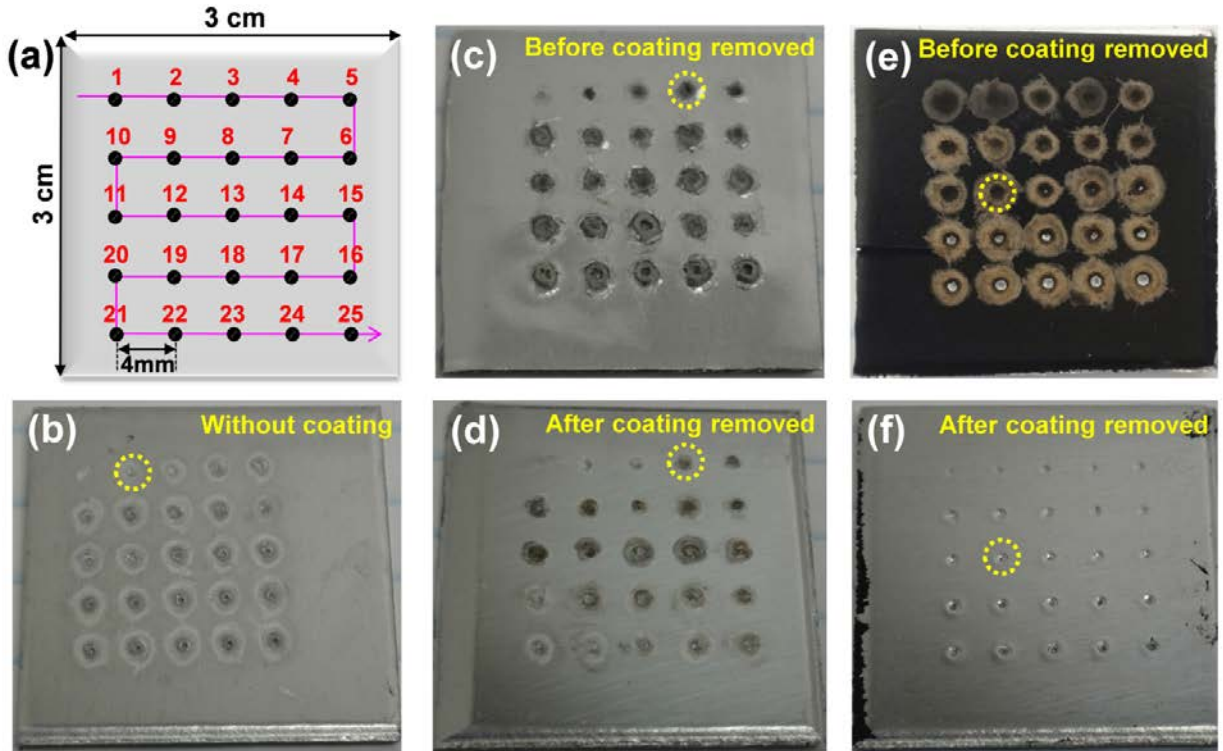


Figure 4. (a) Schematic of the point distribution with different LSP impacts. (b-f) Laser-peened 5052 Al alloy samples (b) without sacrificial coating, using (c,d) Al foil tape and (e,f) black tape as the sacrificial coatings.

3.2.1.1 The effect of sacrificial coatings on laser peening

The use of absorbent sacrificial coatings is found to increase the shock wave intensity in addition to protecting the sample's surface from laser ablation and melting. Therefore, it is very important to select effective, practical sacrificial coatings for LSP applications.

In Fig. 4a, the schematic of LSP-impacted points on Al alloy samples with different impact times, from 1 to 25, is shown. The distance between nearby impacted points is about 4 mm, which could avoid the interactions among different points. Figures 4b to 4f show the optical images of a laser-peened 5052 Al alloy sample plate (b) without sacrificial coating, using (c,d) Al foil tape and black tape as the sacrificial coatings, respectively. Obviously, increasing LSP impact times could result in a higher possibility of laser damage to the sample surfaces.

The dashed yellow circles used in Fig. 4b to 4f indicate the impacted points from which the penetration of the sacrificial coating or alloy surface damage could be observed. In Fig. 4b, a laser-peened sample plate without any sacrificial coating shows surface damage from two LSP impacts. Figures 4c and 4d show the laser-peened sample with Al foil as the sacrificial coating (c) before and (d) after the coating was removed, respectively. The penetration of the coating in Fig. 4c and the damage to the alloy surface in Fig. 4d both begin from four LSP impacts. The laser-peened samples with black tape as the sacrificial coating, before and after the coating was removed, are shown in Fig. 4e and 4f, respectively. However, under this condition, the penetration of coating and the damage of the alloy surface started from 12 LSP impacts, which is much higher than with Al foil. Apparently, the black tape could endure more laser impacts than Al foil, while it is easier for the LSP to damage the sample surfaces without sacrificial coatings.

The surface damage induced by laser direct irradiation should be avoided during the LSP process, which could increase the surface roughness, decrease the residual stress, and reduce the corrosion resistance. On the other hand, higher impact times are preferred to generate higher residual stress and better corrosion resistance. Thus, the black tape is considered to be a preferred sacrificial material and will be used in further experiments.

3.2.1.2 The effect of LSP impact time on the properties of 5052 Al alloy

A. Surface roughness and surface profile

Scanning probe microscopy (SPM), SEM, and a 3D profiler were used to characterize the surface roughness and profile variations of the laser-peened 5052 Al alloy. As shown in Fig. 5, SPM (5a-5d) and SEM (5e-5h) images indicate that the surface roughness increased slightly as LSP impact times increased, which was inevitable for the LSP process and will not seriously affect the residual stress and corrosion resistance. On the other hand, the 3D surface topographies (5i-5l)

reveal that the depth of the laser-peened area (dents) increases proportionally with higher impact times. With one, two, and three LSP impacts, the dent depths are 13, 24, and 42 μm , respectively. This result indirectly indicates higher hardness and residual stress of the dents with increasing impact times.

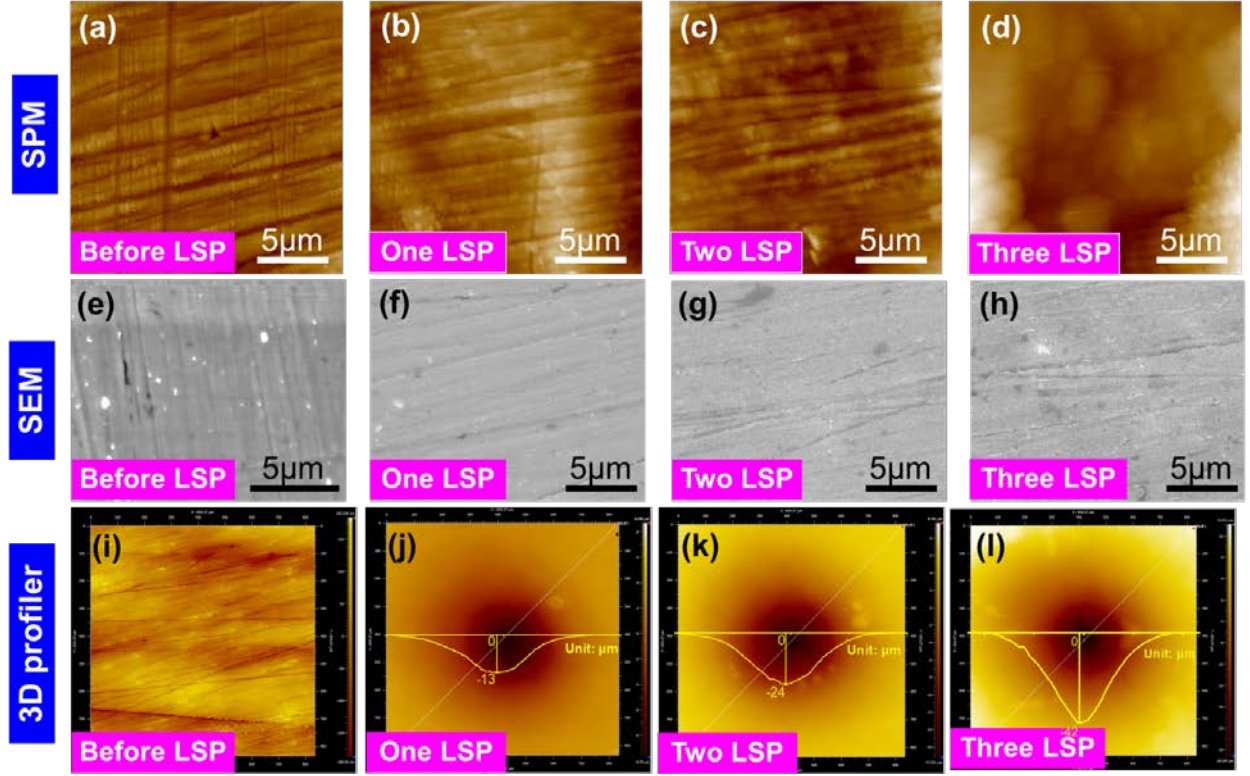


Figure 5. (a-d) SPM images of 5052 Al alloy surfaces treated by a single pulse LSP for (a) zero, (b) one, (c) two, and (d) three impacts. Corresponding (e-h) enlarged SEM images and (i-h) 3D surface topographies of the 5052 Al alloy are shown in (a-d). Insets in (j, k, l) show the cross-sectional height profiles along the lines in the same images.

B. The effect of LSP on surface nanohardness and elastic modulus

Nanoindentation has been established as a powerful technique to characterize mechanical properties at small scales. The force required to press a sharp diamond indenter into the samples tested is recorded as a function of indentation depth. Both elastic modulus and nanohardness can be extracted directly from the nanoindentation curve. Elastic modulus and nanohardness are determined based on the knowledge of the tip shape function and the load-displacement curve (load F and displacement H) [4]. Figure 6d shows the typical load-displacement curve of 5052 Al alloys without LSP.

The nanohardness H and elastic modulus E_r were defined as follows:

$$H = \frac{P_{max}}{A} \quad (3.1)$$

$$E_r = \frac{\sqrt{\pi} S}{2\beta\sqrt{A}} \quad (3.2)$$

where P_{max} is the maximum load, A is the projected contact area, S is the banner slope of the unloading curve, and β is the contact compliance between the indenter and the sample, which is equal to the tangent to the force-displacement curve during unloading after correction for frame compliance. The symbol A in the above equations is calculated as:

$$A = 24.56h_c^2 + \sum_{i=0}^7 C\beta h_c^{1/2^i} \quad (3.3)$$

$$h_c = h - \beta P_{max}/S \quad (3.4)$$

where h_c is the contact depth and h is the indentation depth.

Figures 6a-c show the representative in situ SPM images of 5052 Al alloy surfaces (a) before and (b,c) after nanoindentation, where obvious residual indentations were formed after nanoindentation, indicating residual indent impression. Figures 6d-f show the measured load-displacement curves of the 5052 Al alloy at a maximum load of 10000 μ N treated by single pulse laser peening for (d) zero, (e) one, and (f) two impacts, respectively. For each condition, the measurements were repeated five times; and an average value was determined on the basis of the five measured data. The corresponding calculated experimental data (surface nanohardness, elastic modulus, contact depth) are shown in Fig. 7. From Fig. 7a, it can be seen that the contact depths of the indenter were 602 ± 13 , 527 ± 13 , and (369 ± 7) nm in the regions by LSP for zero, one and two impacts, respectively. The maximum contact depths of the indenter decreased rapidly from the non-laser-peened region to the laser-peened region and decreased further with the increase in laser impact times (Fig. 7a).

The measured value of surface nanohardness for different laser impacts is shown in Fig 7b. It can be seen that the value of surface nanohardness in the laser-peened region obviously improved in comparison to the corresponding value in the non-laser-peened region. Surface nanohardness reached about 1.47 GPa in the laser-peened region with a single impact, which improved by 15.9% over that in the non-laser-peened region (1.26 GPa). When the laser impact was increased to twice on one spot, the surface nanohardness value reached 2.93 GPa, which was larger by 133% than in the non-laser-peened region.

The dependence of elastic modulus on LSP impacts is shown in Fig. 7c. The value of elastic modulus was 47.7 GPa in the non-laser-peened region, and the corresponding values in the regions with single and two LSP impacts were 166.3 and 304.0 GPa, respectively. The value of

the elastic modulus in the regions with single and two LSP impacts increased by 249% and 537% compared to that in the non-laser-peened region, respectively.

The above analysis indicates that both the values of surface nanohardness and elastic modulus in the laser-peened region were obviously larger than those in the non-laser-peened region and increased with the increments of LSP impacts. Although the elastic modulus is an intrinsic property of the material and fundamentally relates to the atomic bonding, the elastic modulus can be changed by some surface treatment technologies [5]. It is well known that increasing the elastic modulus is advantageous in enhancing stiffness of samples, which decides the stability when the components service harsh environments [6]. Therefore, it can be concluded that LSP can improve the stiffness of the Al alloy.

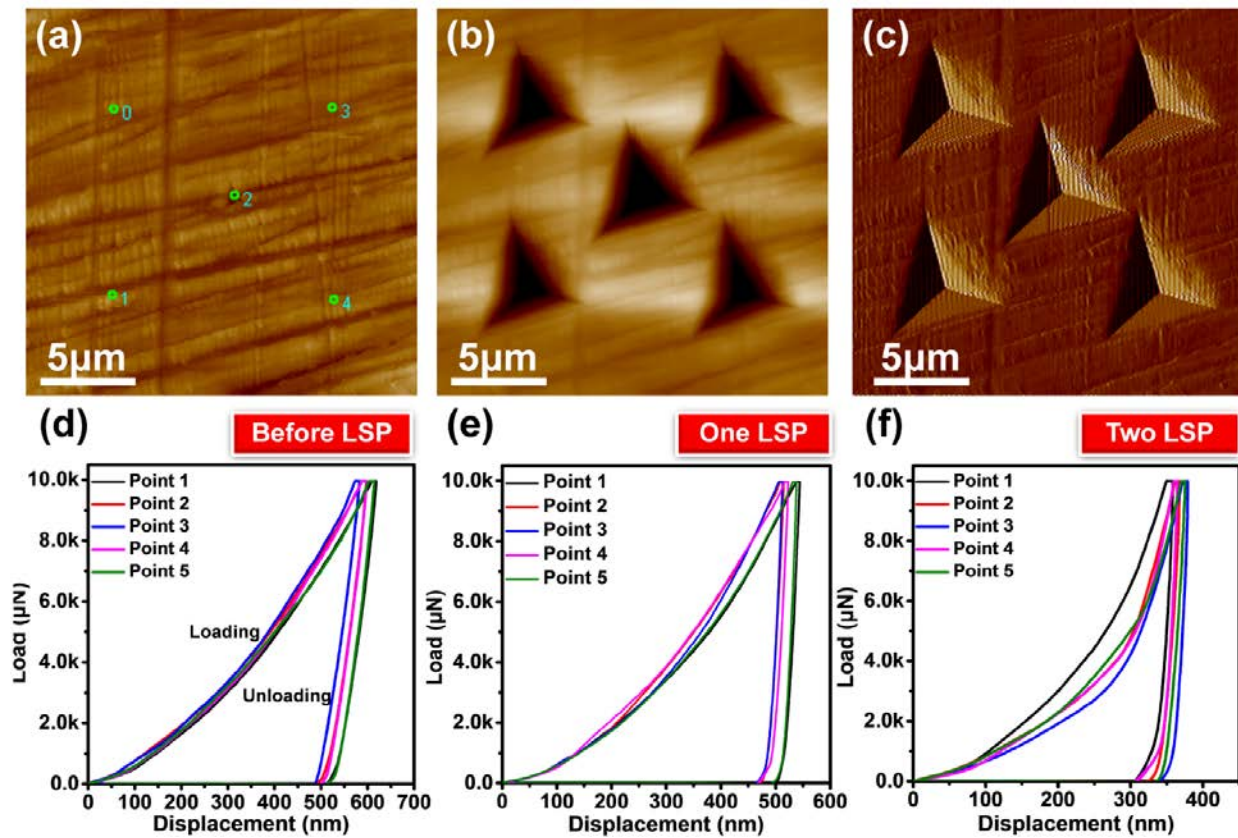


Figure 6. (a) Representative SPM image of 5052 Al alloy surfaces before nanoindentation. (b,c) Corresponding and in situ SPM (b) morphological and (c) phase images of the 5052 Al alloy after nanoindentation. (d-f) The measured load-displacement curves of the 5052 Al alloy treated by single pulse LSP for (d) zero, (e) one, and (f) two impacts.

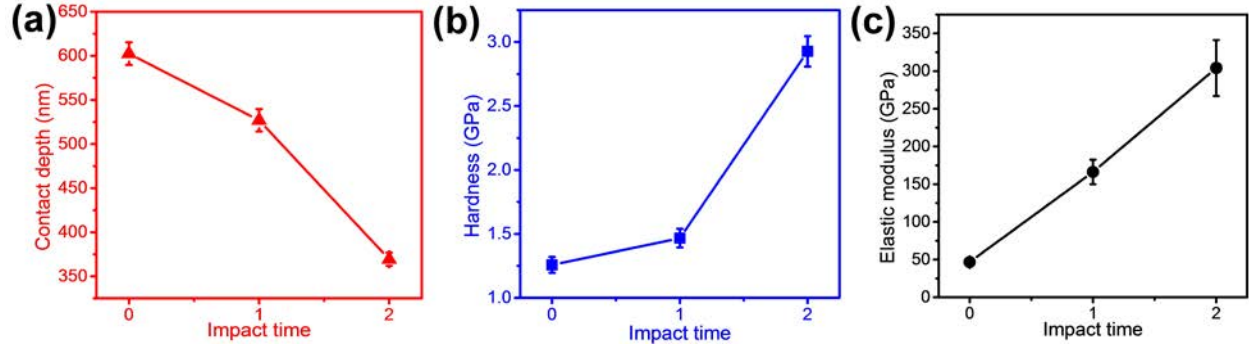


Figure 7. The profiles of the (a) contact depth, (b) surface nanohardness, and (c) elastic modulus of 5052 Al alloy as a function of laser peening impacts.

3.3 Conclusions

In the first reporting period of this project, the experimental setup for laser peening was developed in our lab. The effectiveness of our laser peening system was tested using 5052 Al alloy for the samples tested. The effects of LSP processing parameters on the surface morphology, nanohardness, and elastic modulus of 5052 Al alloy were mainly investigated and analyzed. Some important conclusions have been summarized as follows:

- (1) The experimental setup for the LSP system has been successfully developed. This LSP system can meet the demands of single and multiple LSP impacts. The laser source is a continuum Q-switched Nd:YAG PR II 8010 laser. The laser pulse frequency can be changed from 1 to 10 Hz. The normally used pulse duration of this laser is approximately 6-8 ns, and the maximum pulse energy is 800 mJ with a wavelength of 1064 nm. The output diameter of the laser beam from this laser is about 5 mm. Two flat Nd:YAG mirrors and a flat convergent lens (focal length: 20 cm) were used to deliver the pulse produced by the laser. A flowing water film with a layer thickness of 1-2 mm was used as a transparent confining medium. Black tape with a thickness of 177 μm was optimized as a sacrificial layer to protect the sample surface from the thermal effect.
- (2) With appropriate processing parameters, macro plastic deformations (dents) can be formed on the sample surfaces through ns laser-induced shock waves. Surface morphologies and 3D profiles of dents were measured. Both surface roughness and dent depth increased with increased LSP impact times.
- (3) For 5052 Al alloy samples, the contact depths for the laser-peened samples were lower than those for the non-laser-peened samples; the values of surface nanohardness and elastic modulus for the laser-peened samples were obviously larger than those for the non-laser-peened samples. In addition, the values of surface nanohardness and elastic modulus increased with the incremental increase in LSP impact times.

4. Description of Any Problems/Challenges

No problems were experienced during this reporting period.

References

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OFFICE OF SPONSORED PROGRAMS
Post-Award Administration
151 Whittier Research Center, 2200 Vine Street
Lincoln, NE 68583-0861
FED ID # 47-0049123

AGREEMENT TITLE/CONTRACT NUMBER:
N00014-15-C-0087
Portable Fiber Laser System

INVOICE/REPORT: 2511090140-01

INVOICE PERIOD: 5/15/2015 to 7/31/2015

Directed by Dr. Yongfeng Lu

REFERENCE NUMBER: 25-1109-0119-001

ANALYSIS OF CLAIMED CURRENT AND CUMULATIVE COSTS

MAJOR COST ELEMENTS	APPROVED BUDGET	AMOUNT FOR CURRENT PERIOD	CUM. AMOUNT FROM INCEPTION TO DATE
Salaries and Wages	\$227,566.00	\$1,739.13	\$1,739.13
Employee Benefits	\$76,488.00	\$32.00	\$32.00
Operating	\$1,250.00	\$0.00	\$0.00
Consumable Supplies	\$32,402.00	\$0.00	\$0.00
Travel-Domestic/Foreign	\$10,000.00	\$0.00	\$0.00
Equipment	\$35,000.00	\$0.00	\$0.00
Indirect Cost	\$167,293.00	\$903.28	\$903.28
Total Amount	\$549,999.00		\$2,674.41
	Current period expenses	\$2,674.41	

ANALYSIS OF CLAIMED CURRENT AND CUMULATIVE COSTS

"I certify that all expenditures reported are for appropriate purposes and in accordance with the agreement noted above."

Date:

August 27, 2015

Deb Arent - Assistant Director

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